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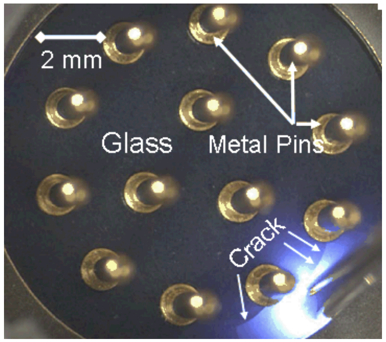

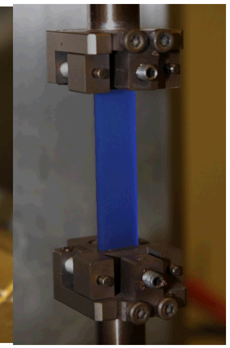

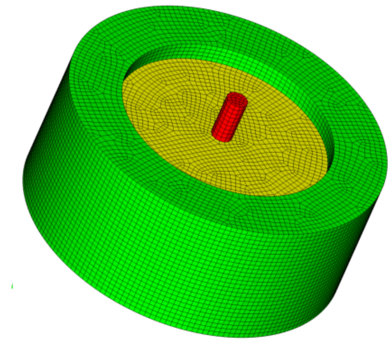

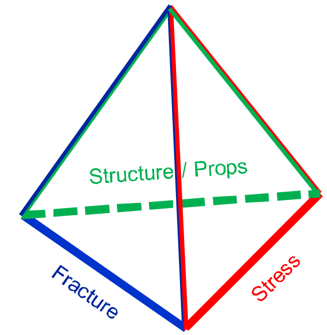

Characterizing and Predicting Stress and Structural Relaxation in Glass

[a]Kevin T. Strong Jr, [b]Brenton Elisberg, [b]Ryan D Jamison, Tom Buchheit and [c]Kevin G Ewsuk

FFAG7

Monday, July 3, 2017

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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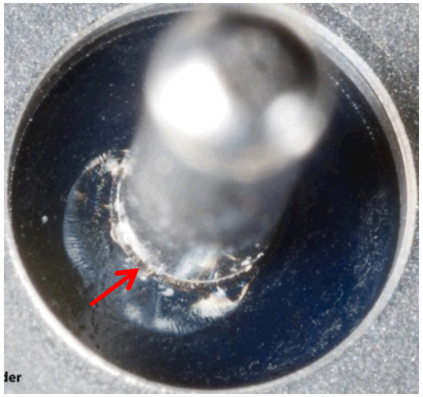
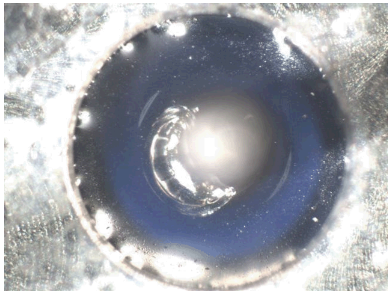
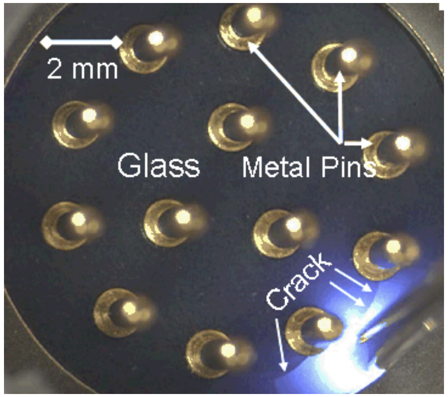
Outline

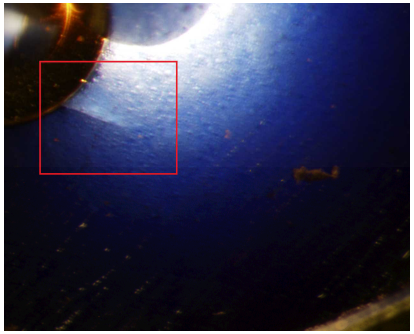
- Motivation
- Approach
- Model Calibration Experiments
- Model Validation
- Current Applications
- Conclusions

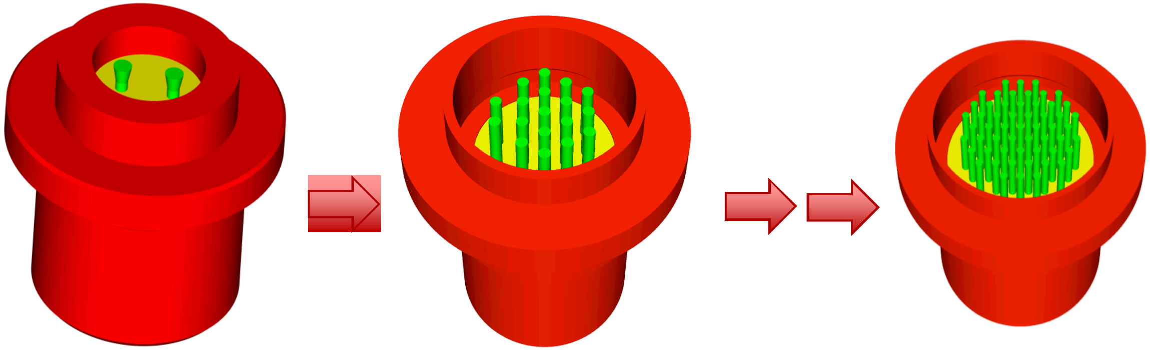
Motivation – Glass to Metal Seals





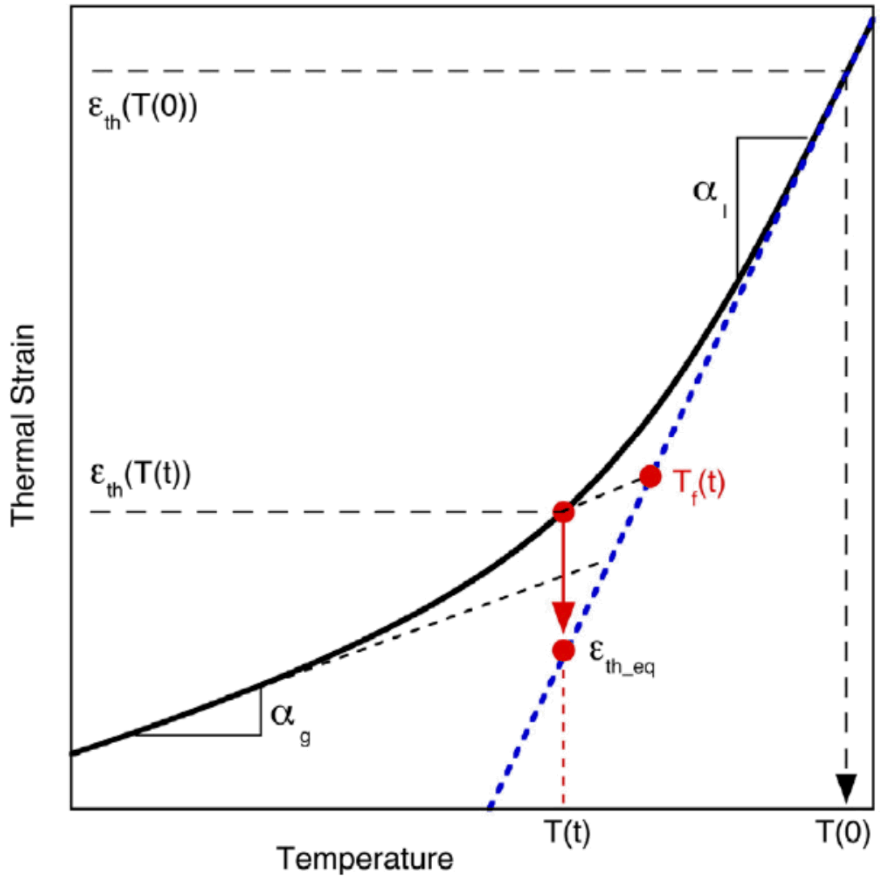






Higher fidelity stress modeling is needed to address shrinking performance margin in more complex designs

Background – Structural Relaxation



Finite Element (FE) Modeling Predicts Residual Stress In Glass-to-Metal Seals

routine
missing

crack initiation

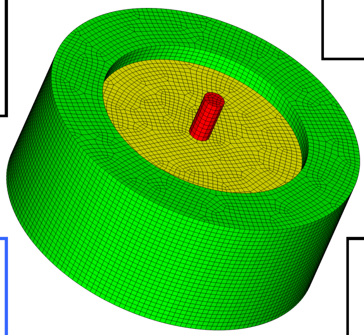
pin plasticity $\sigma_y(T)$, $E_H(T)$

SS small strain

shell plasticity $\sigma_y(T)$, $E_H(T)$

idealized geometry

maximum stress failure criterion



elastic glass

glass transition

bonded interfaces

viscoelastic glass

assumed stress-free temperature, T_{set}

prescribed CTE $\varepsilon_{th}(T)$

history dependent thermal strain

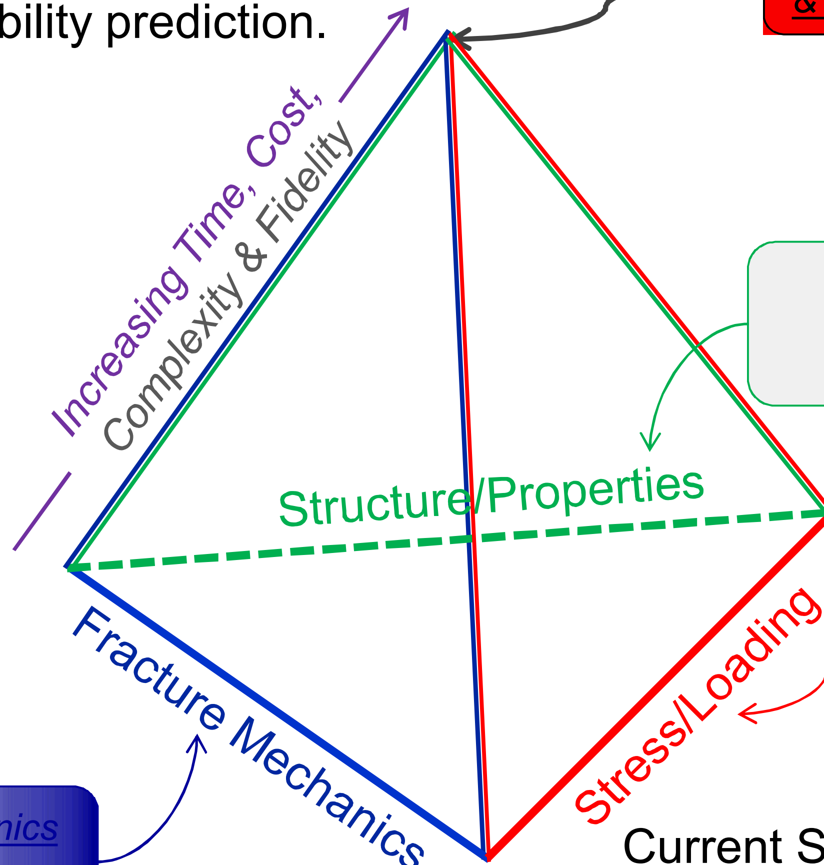
fracture mechanics

Higher fidelity stress modeling is possible with improved materials behavior models

BrittMAPP – Brittle Materials Assurance Predictability Program

Future State (2020): **Quantitative** mechanics-based failure & reliability prediction.

Quantitative brittle failure & lifetime reliability prediction



Determine & model process-*structure-property* relations.

Stress determination considering materials, processing & *loading* history.

Fracture mechanics
Crack initiation, stability, & growth

Current State (2013): **Qualitative** stress-based failure analysis (i.e., engineering judgment).

Simplified Potential Energy Clock (SPEC) Model

Journal of Non-Crystalline Solids 432 (2016) 545–555

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Journal of Non-Crystalline Solids

journal homepage: www.elsevier.com/locate/jnoncrysol

Characterization and calibration of a viscoelastic simplified potential energy clock model for inorganic glasses

CrossMark

Robert S. Chambers [a,*], Rajan Tandon [b], Mark E. Stavig [c]

[a] *Engineering Sciences Center, Sandia National Laboratories, Albuquerque, NM 87185–0346*
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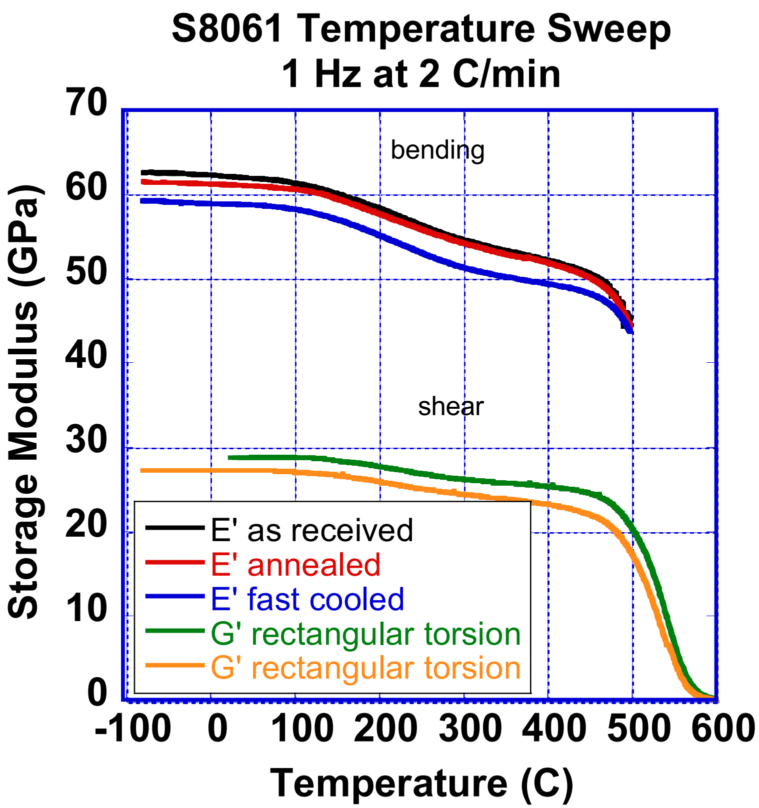
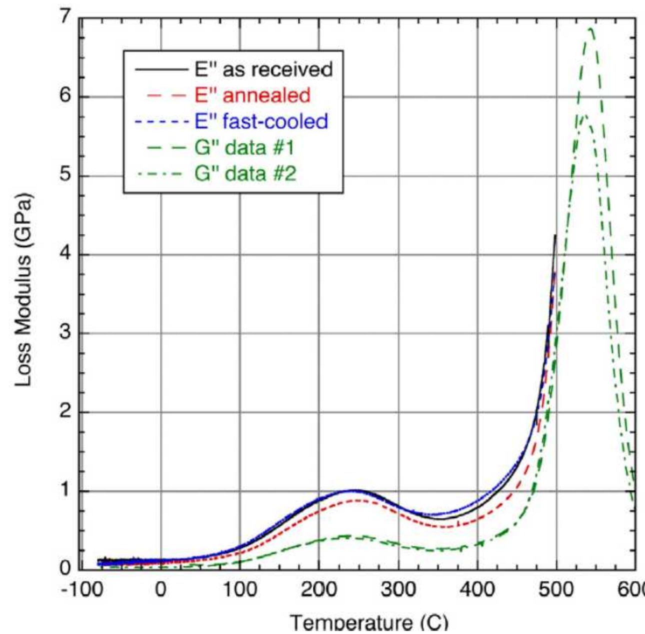
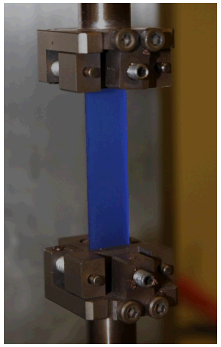
[c] *Materials Science and Engineering Center, Sandia National Laboratories, Albuquerque, NM 87185–0958*

Dynamic Mechanical Analysis Temperature Sweep

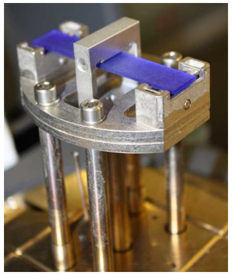




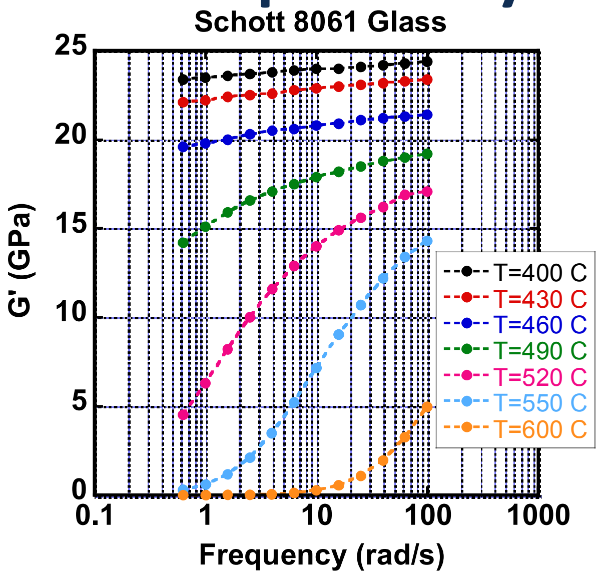
TA ARES Rheometer

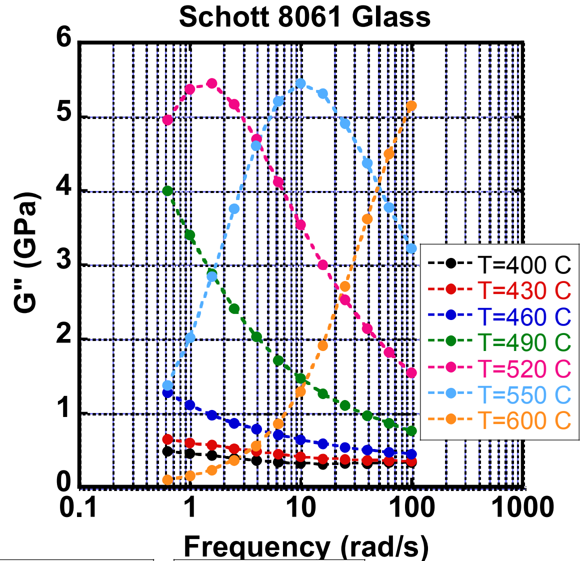


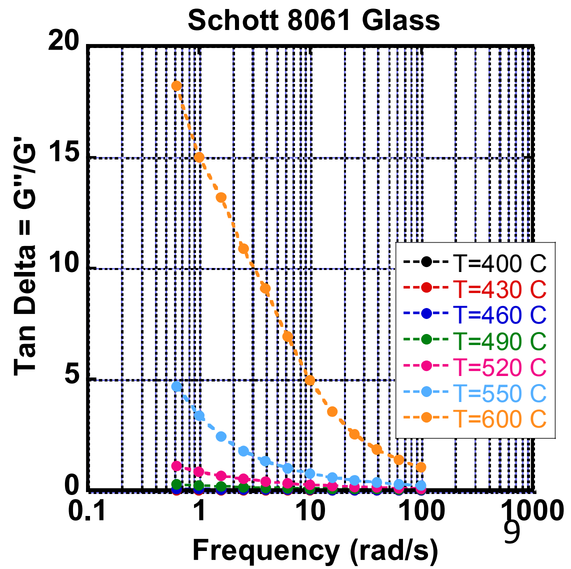
TA Q800 DMA

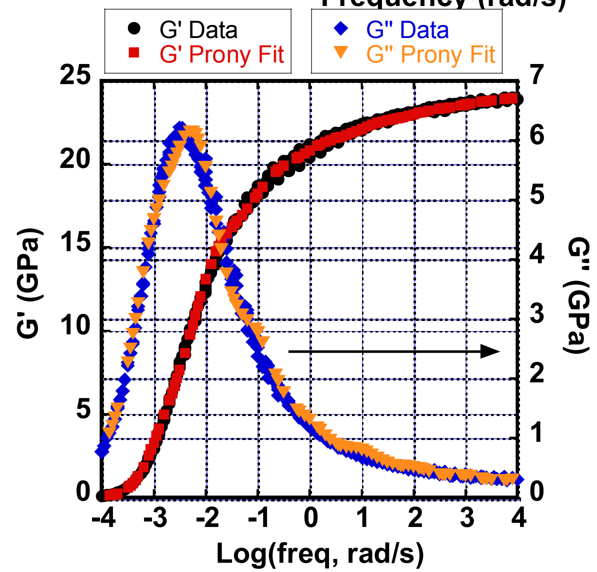


Dynamic Mechanical Analysis Frequency Sweep

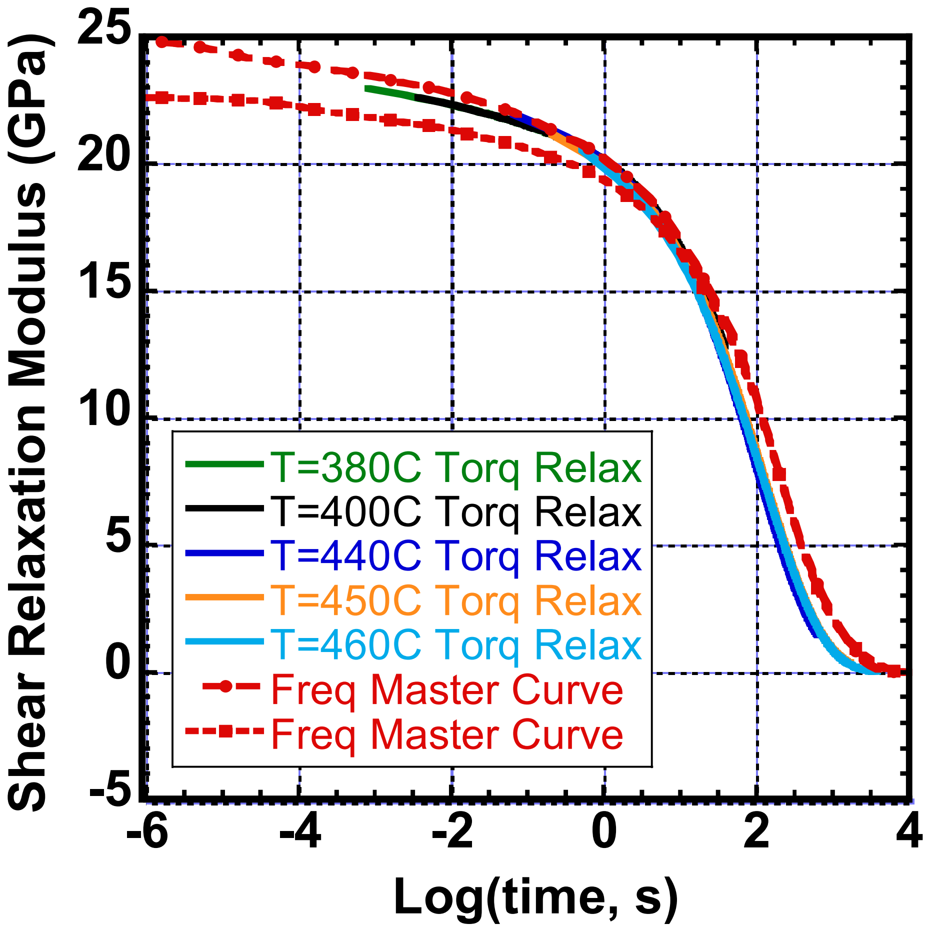




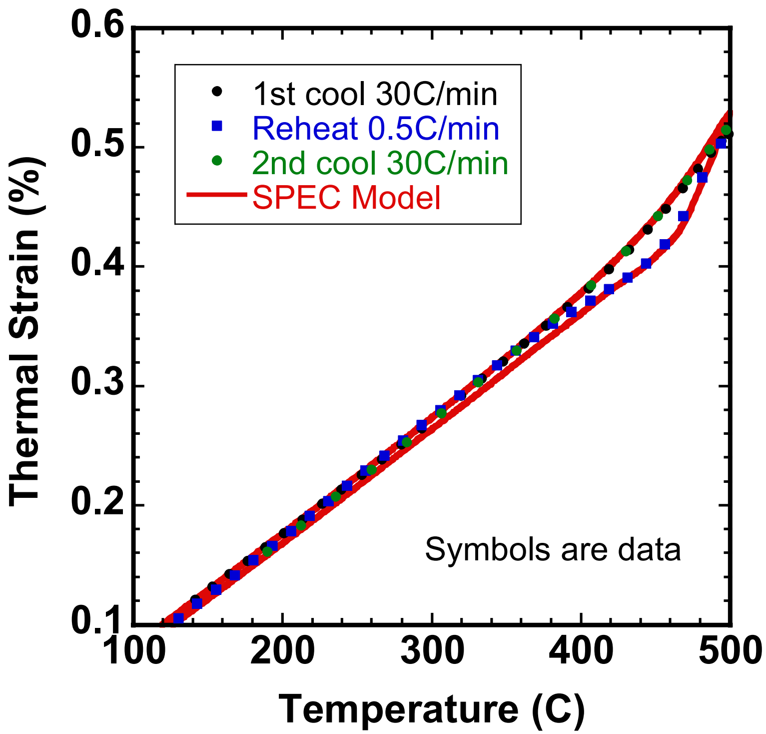


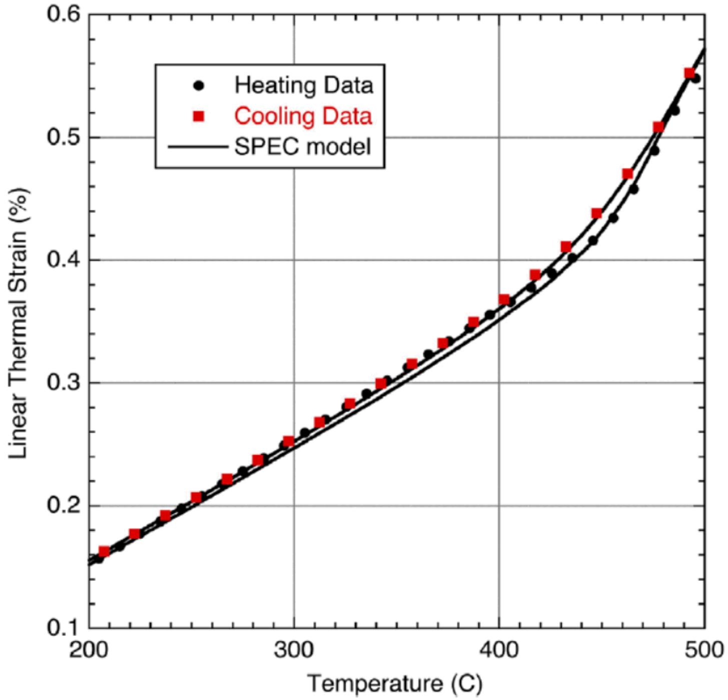


Constructing time based master curve



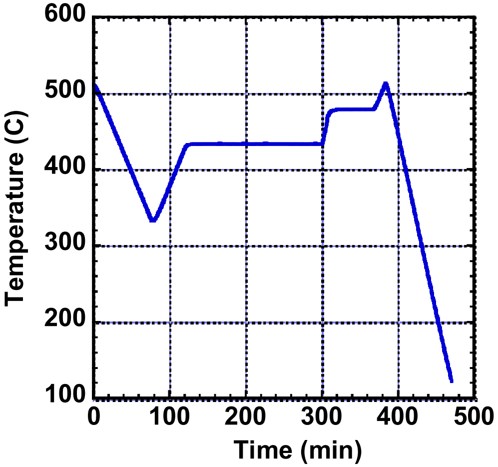
Model Calibration

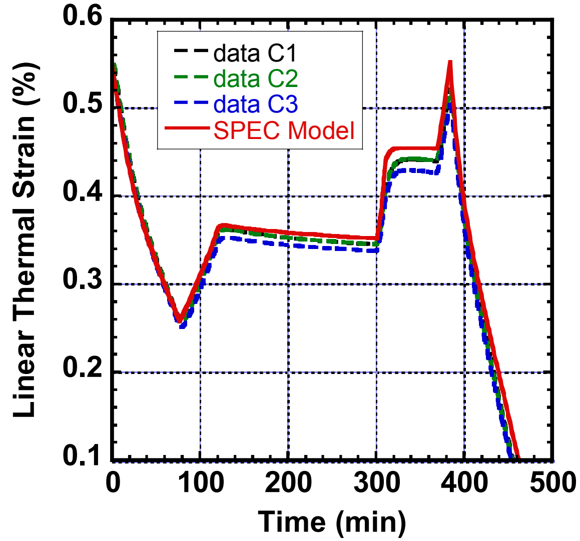


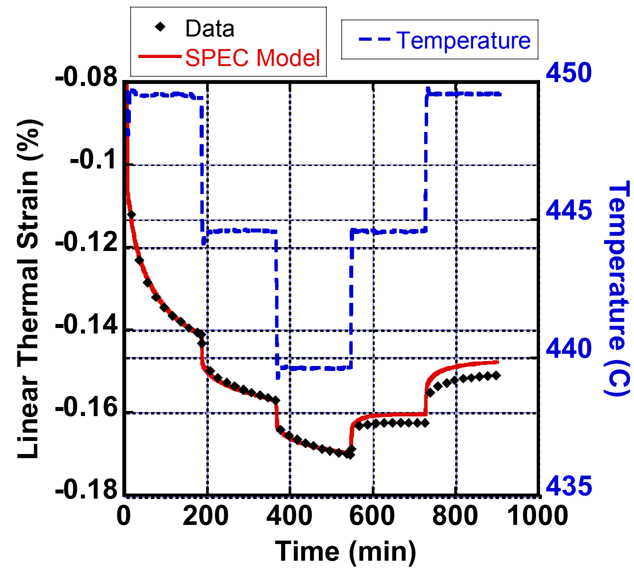


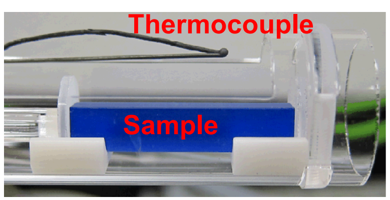
SPEC Model volume response adapted and calibrated to measure glass thermal strain

Model Validation – Complex Thermal History



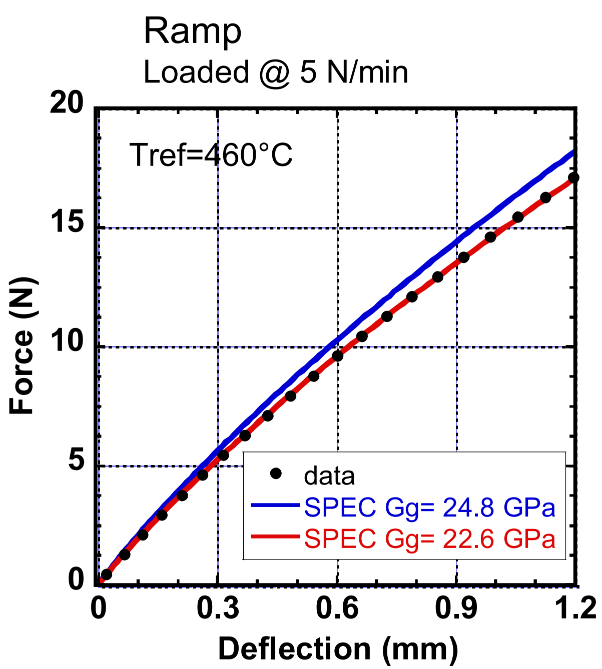




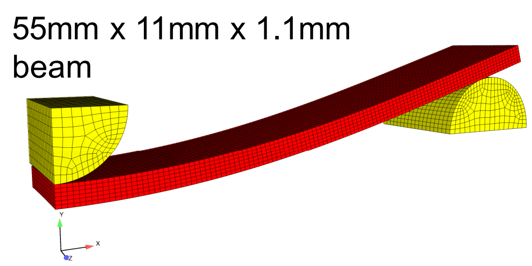


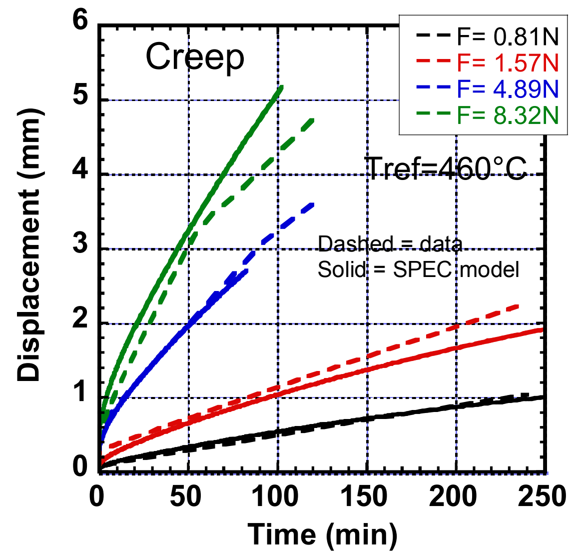
Netzsch Dilatometer

Model Validation – 3 – point bending



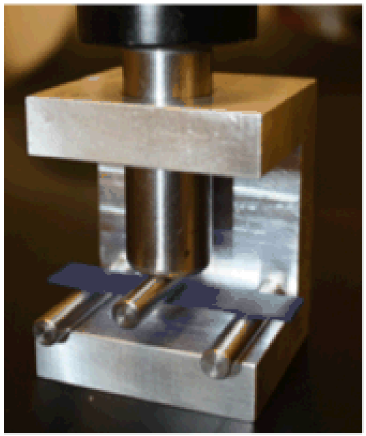
3-Pnt Bending Tests at Tg



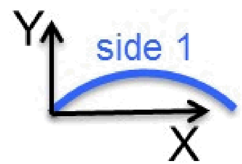


Characterizing Low Temp Creep

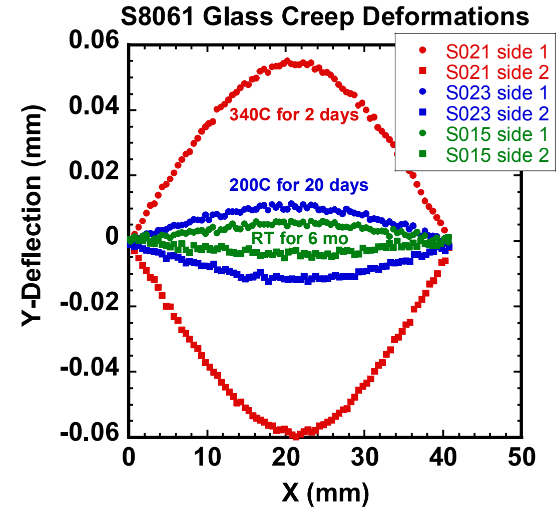
Three-point bend testing



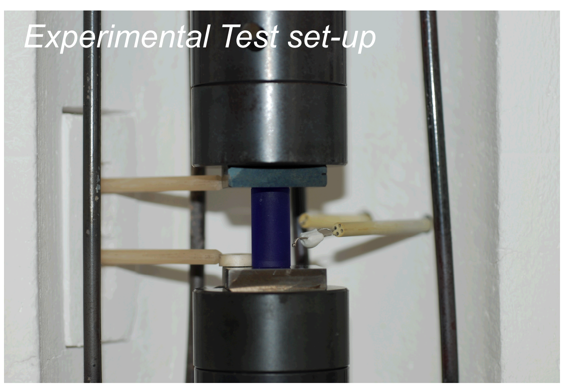
T (°C)	Stress (psi)
340	4220
200	4220
20	4720

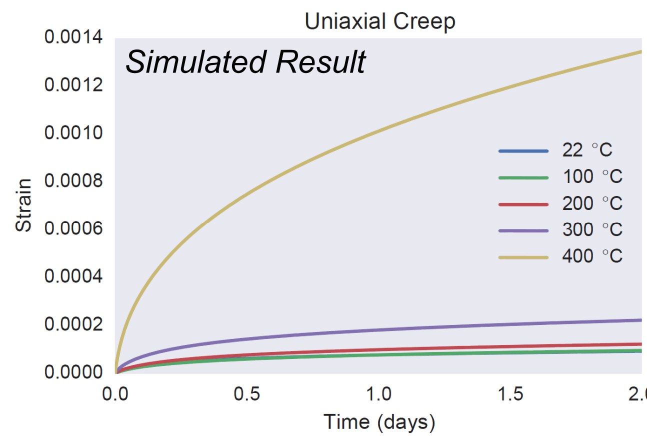


. . . Measurable creep 440°C below Glass Transition



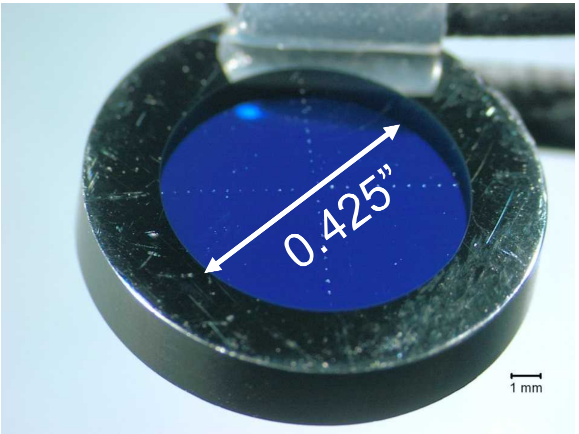
Compression creep testing



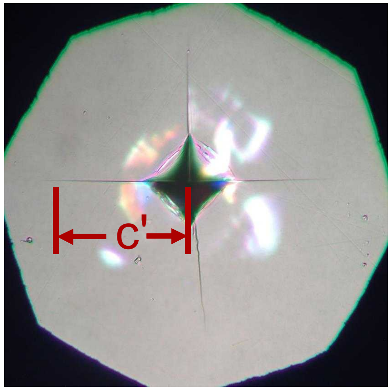


30 MPa

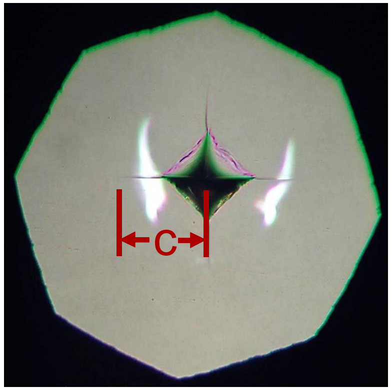
Model Validation: stress mapping by indentation crack measurements



GtoM seal test geometry



Polished Annealed Block
avg. c ≈ 92 um



Indent on test geometry glass (not near SS shell)
avg. c ≈ 55 um

$$K_{Ic}' = \chi\, P/c^{3/2}$$

stressed glass → K_I

unstressed glass → K_{Ic}'

$$\sigma_r = \frac{-(K_I - K_{Ic}')}{1.12\,\pi\, c^{1/2}}$$

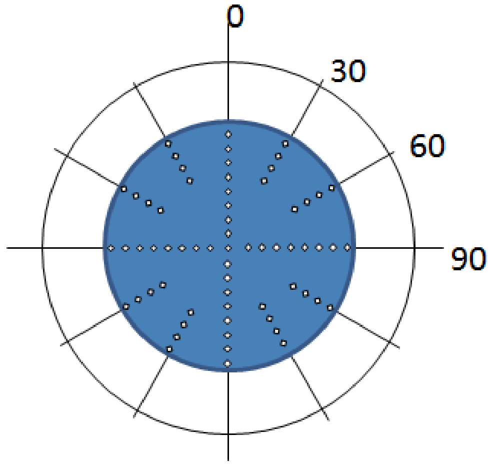
Derived from indentation fracture mechanics concepts

e.g. Lawn Fracture of Brittle Solids (1993)

Model Validation: stress mapping by indentation crack measurements

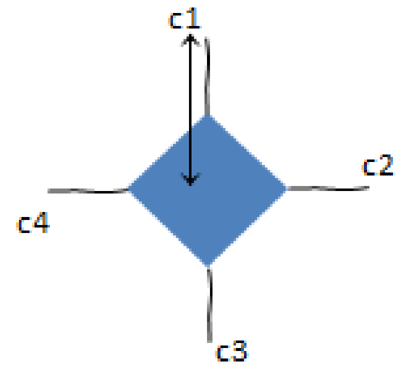
Sandia National Laboratories

Indentation locations

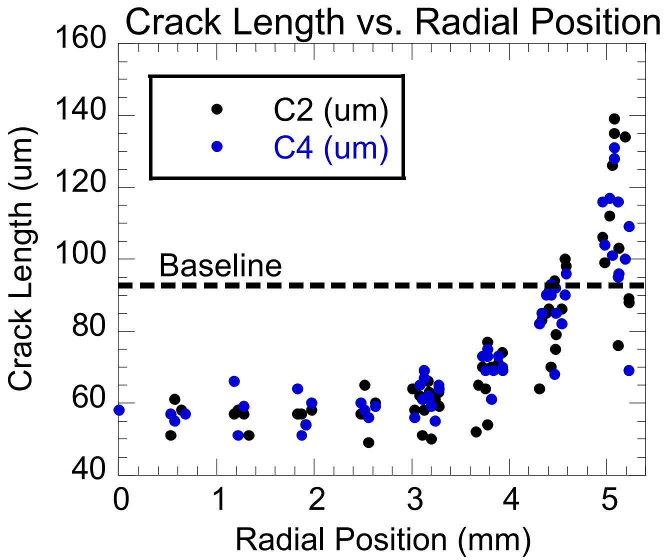


Radial Component
c1 and c3

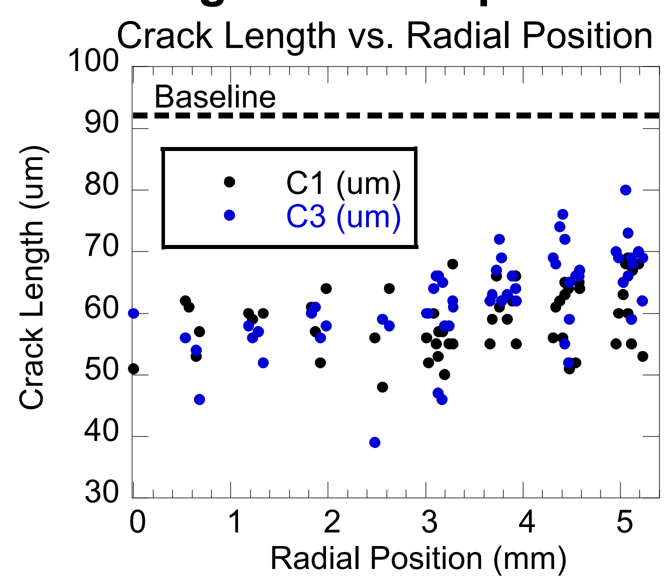
Tangential Component
c2 and c4



Radial Component



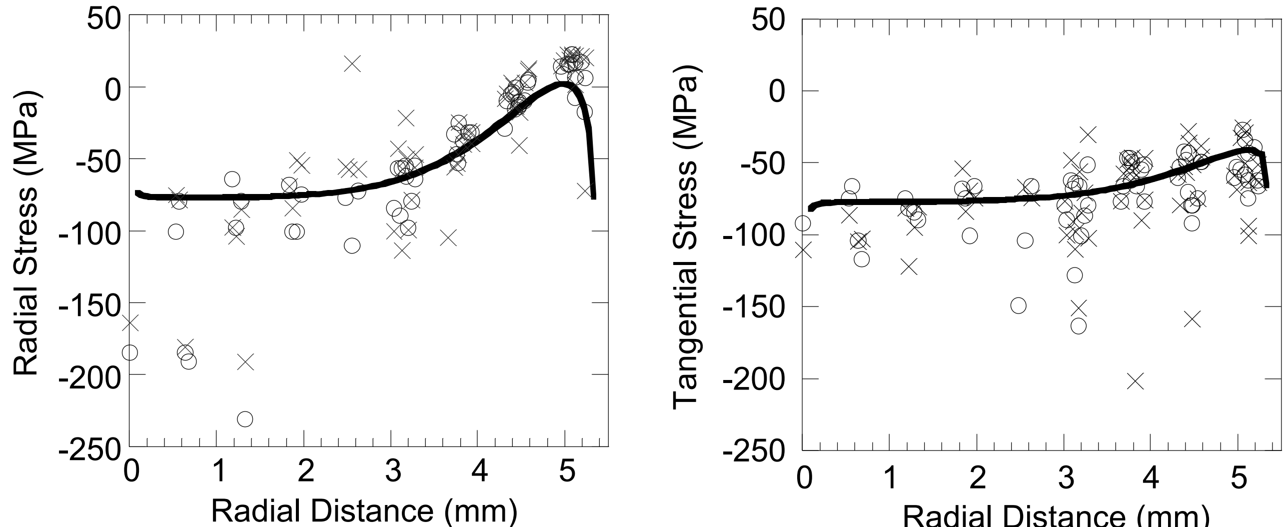
Tangential Component



Measured data has been used to validate analysis

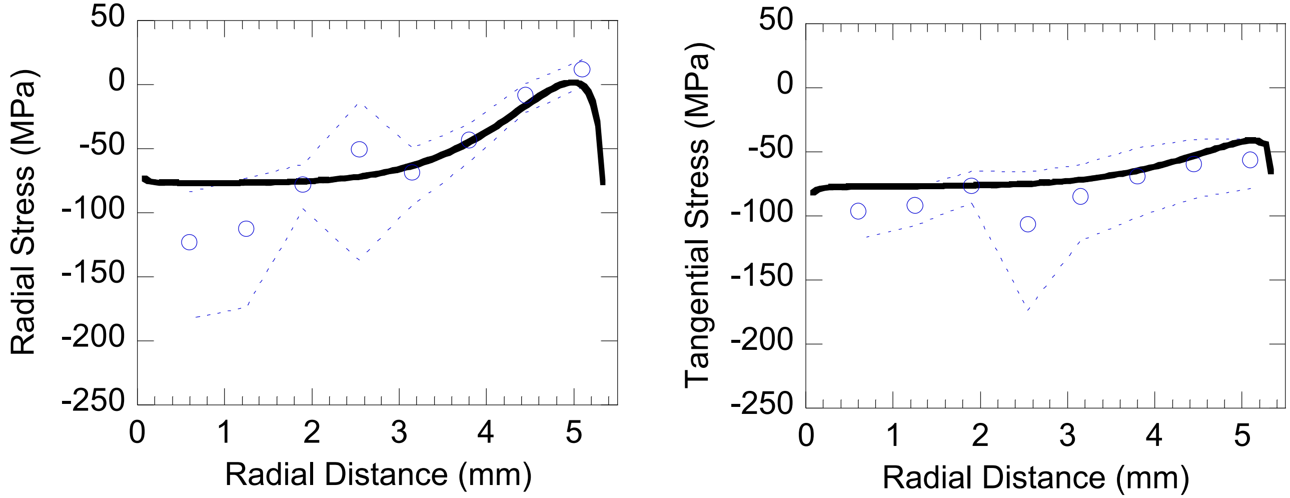
Direct Comparison

○ Measurement #1
× Measurement #2
— BCJ+SPEC model

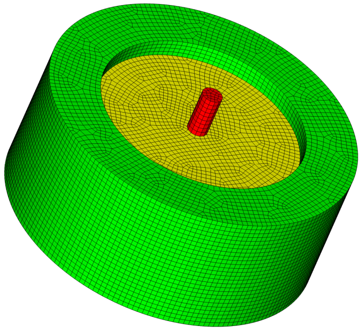


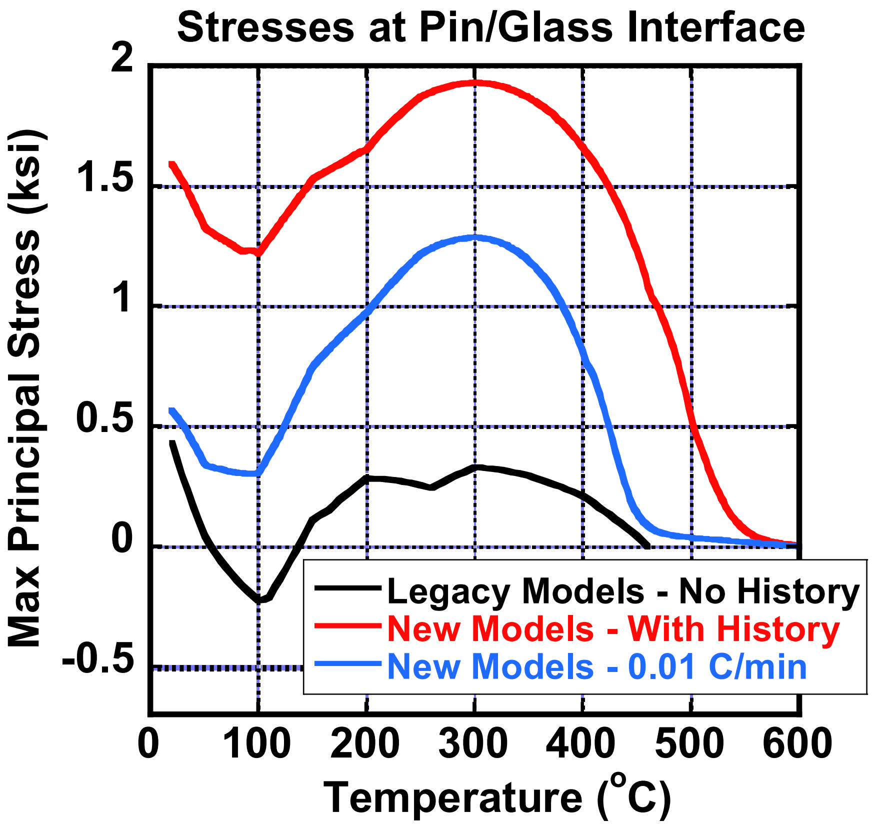
Averaged Result

○ Measurement
— BCJ+SPEC model



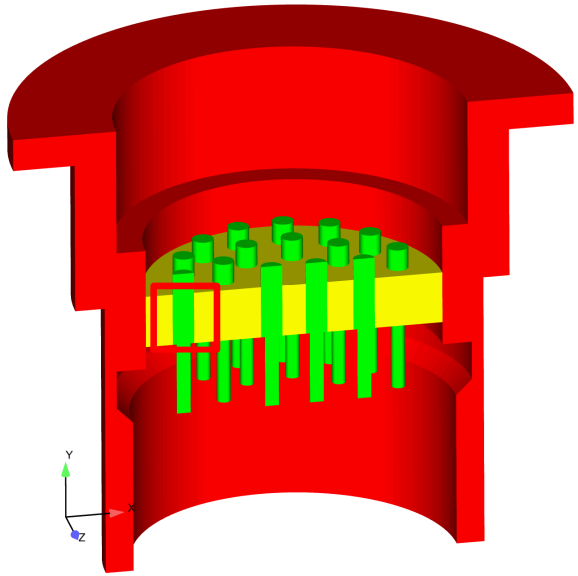
SPEC vs Purely Elastic Model

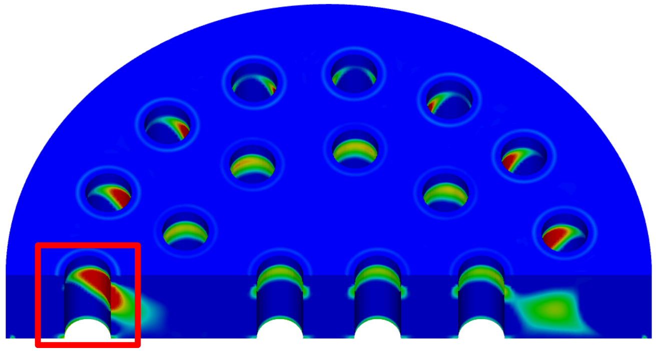


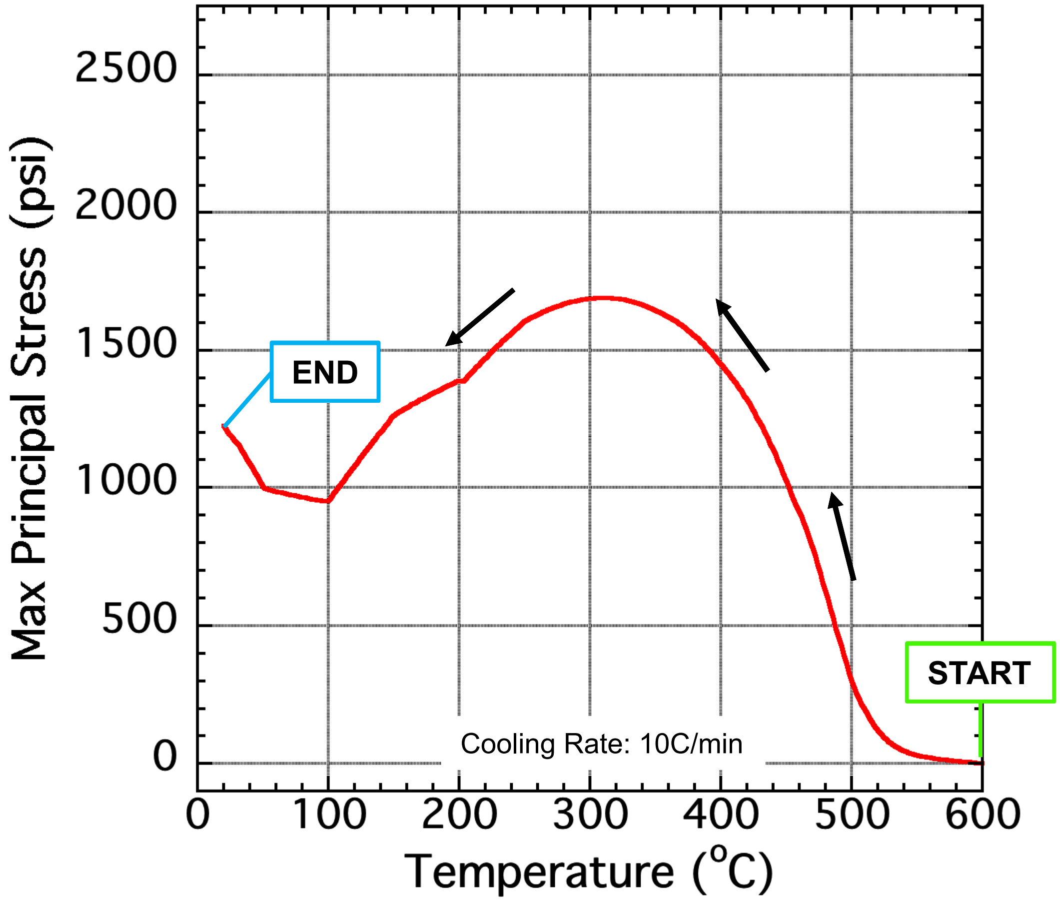


SPEC Model volume response adapted and calibrated to measure glass thermal strain

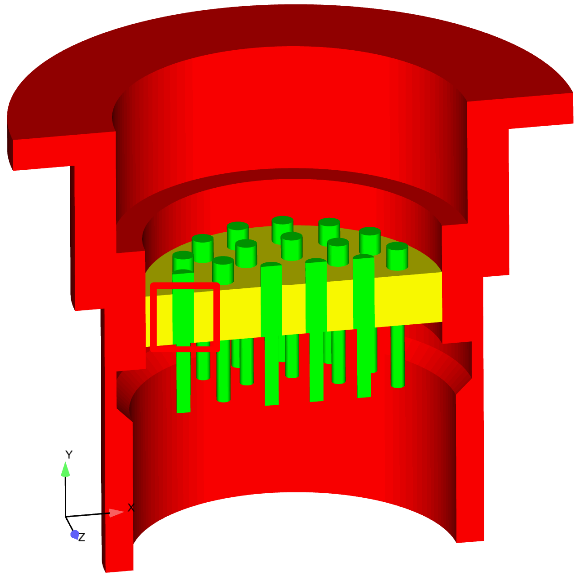
Investigate Complicated Designs

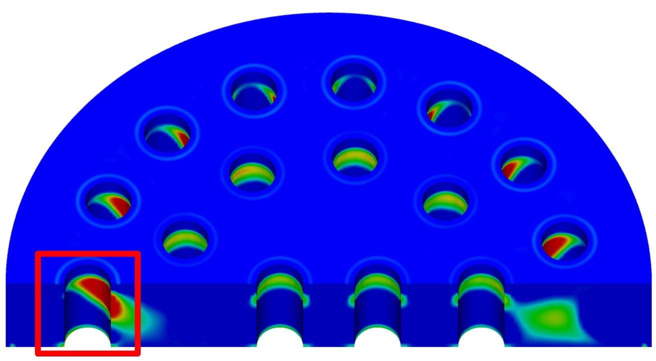


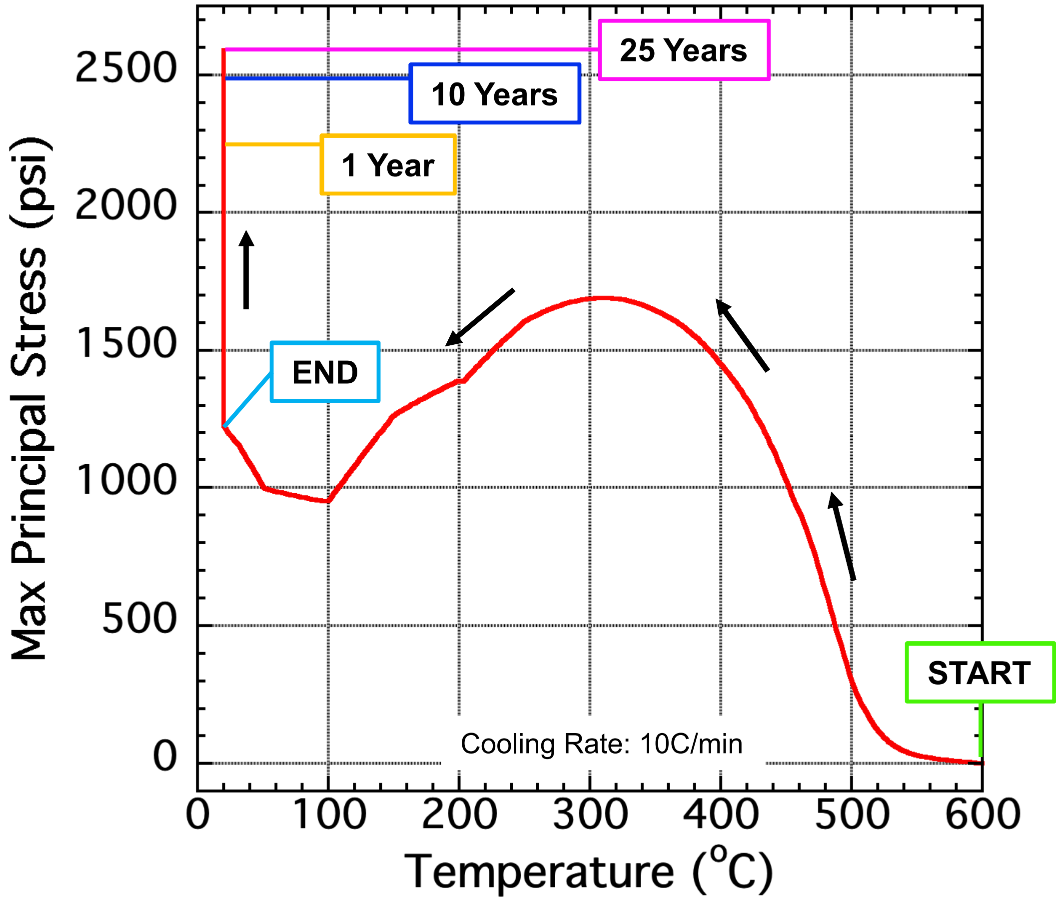




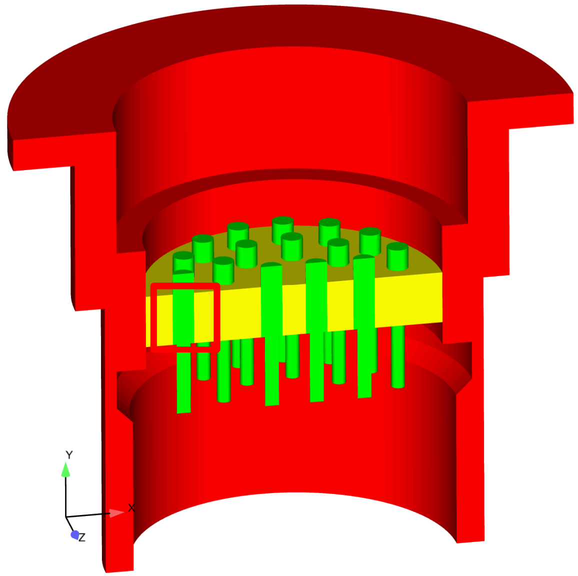
Aging of Compression Seal

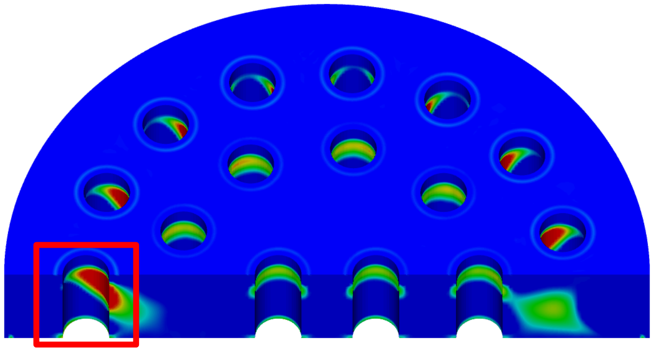


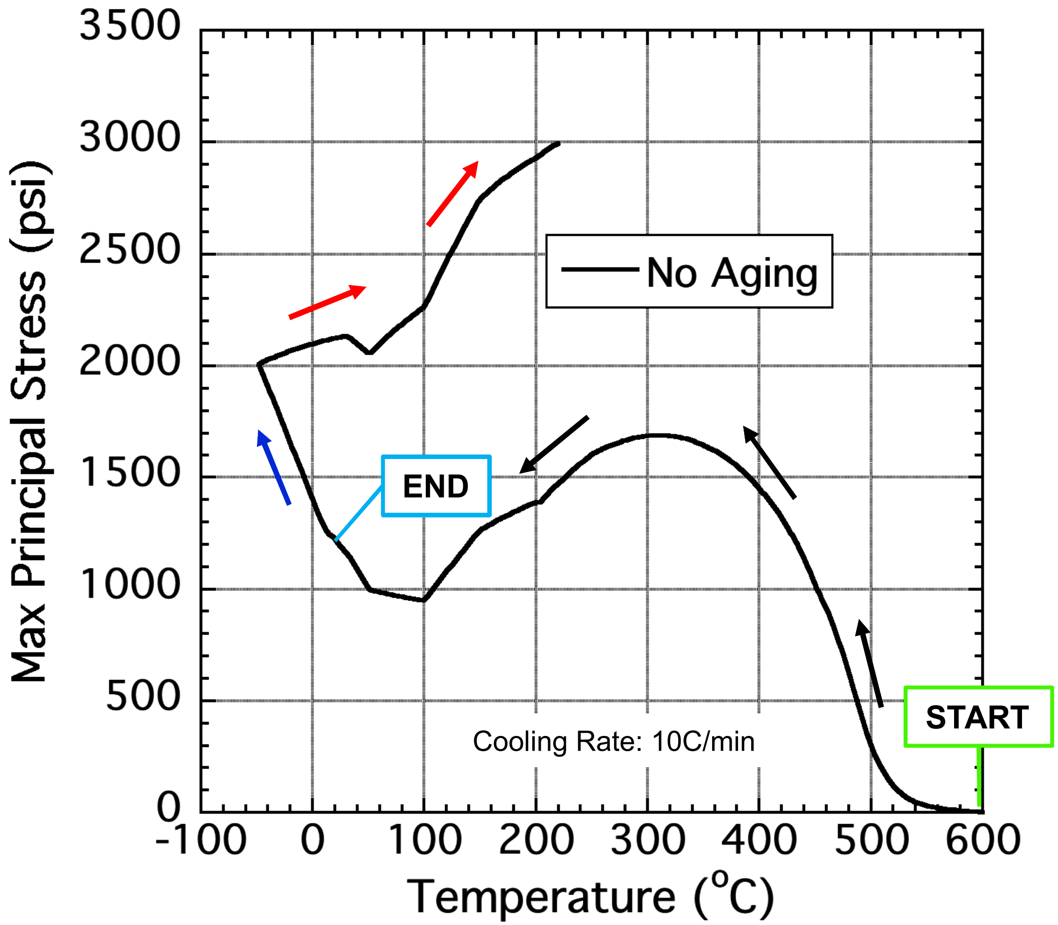




Residual Stress Due to Complex Thermal Histories

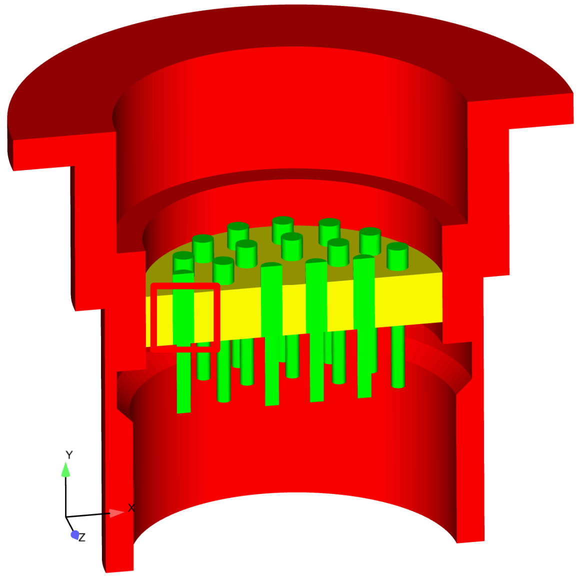


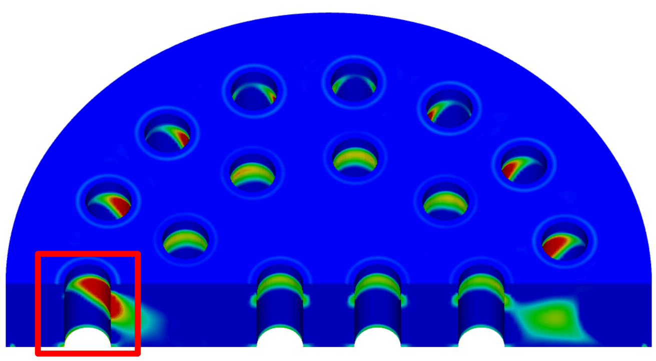


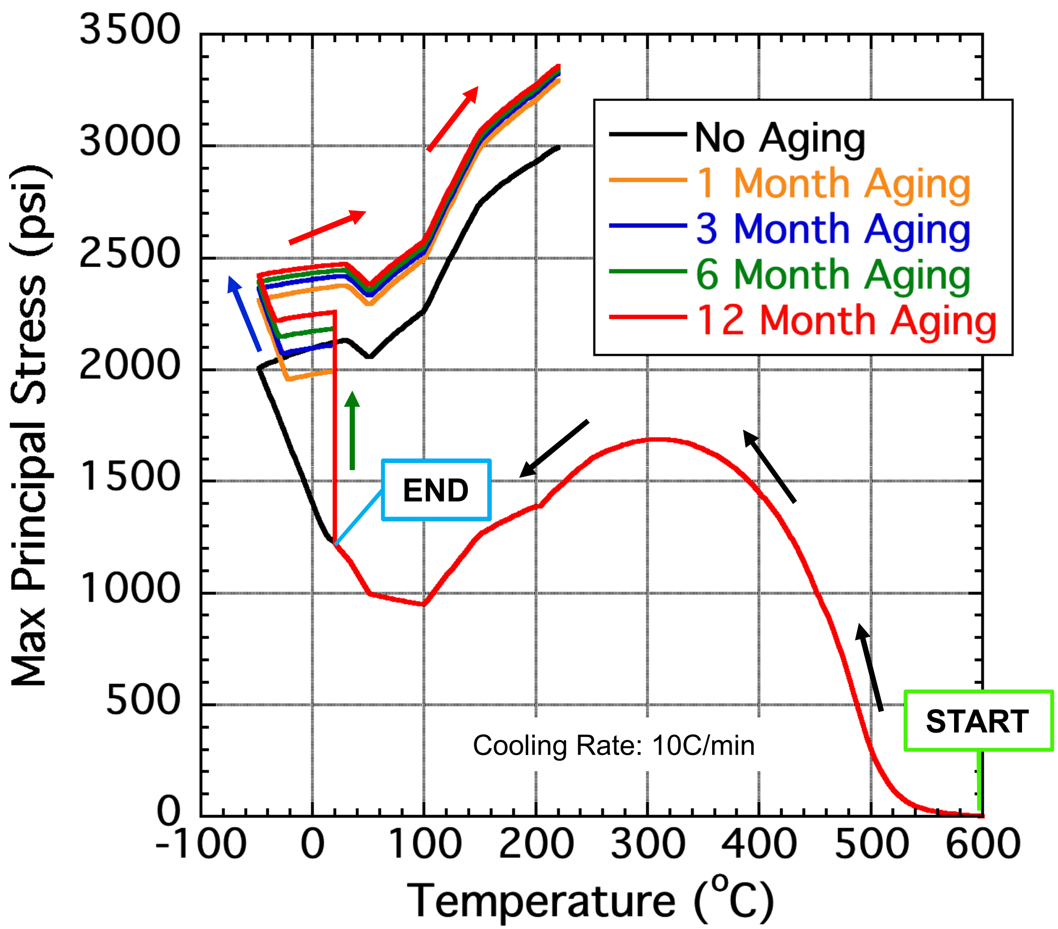


3 °C/min thermal cycle: -50 °C → 220 ° C

Residual Stress Due to Complex Thermal Histories







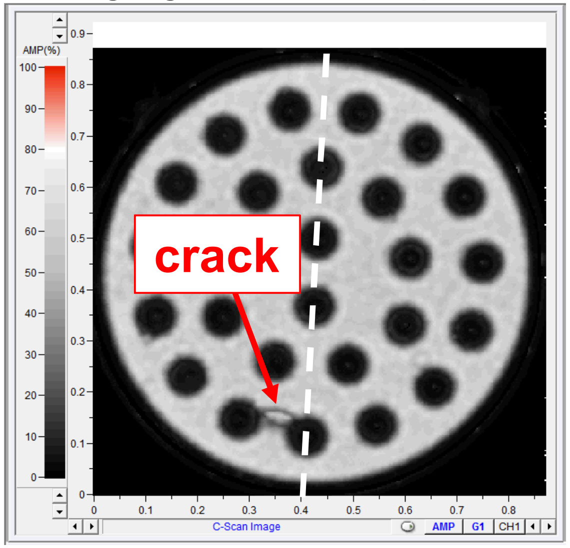
3 °C/min thermal cycle: -50 °C → 220 ° C

Aging Testing

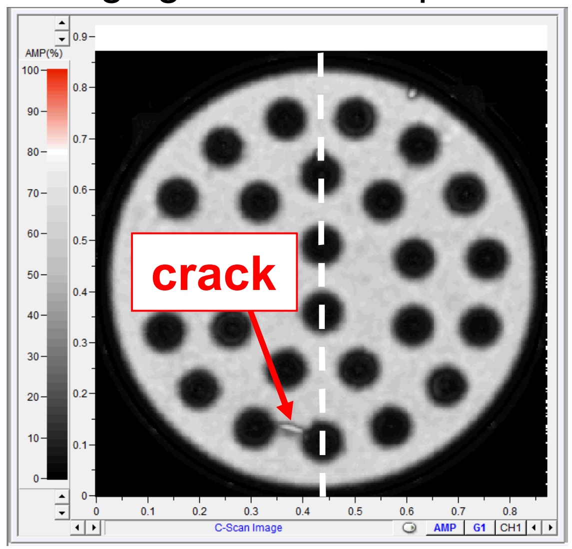
- 10 connectors tested after receiving from supplier
  - Approximately 2 days after manufacturing
- 10 connectors tested 6 months after receiving
- 10 cycles, then ultrasonic scan through glass thickness
  1. -50 °C → 150 °C
  2. -50 °C → 180 °C
  3. -50 °C → 200 °C
  4. -50 °C → 220 °C (oven low/high limits)
- 10/10 connectors tested shortly after receiving did not crack after all thermal cycles.
- 7/10 shells tested 6 months after receiving did not crack after the cycles.
  - **3/10 cracked after the -50 °C → 220 °C thermal cycles.**

Ultra Sound after 220 °C cycle

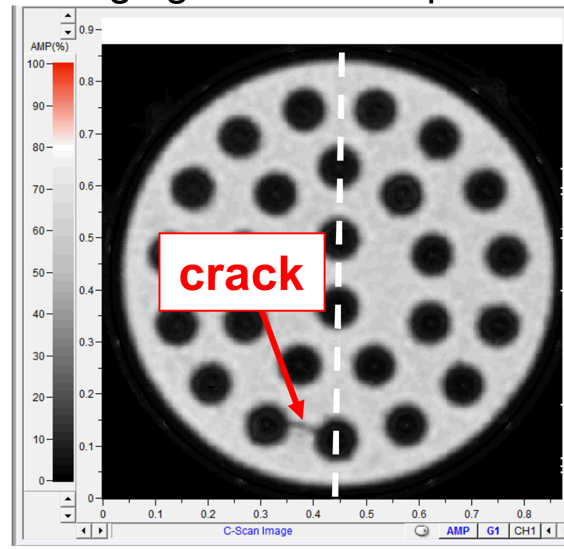
Imaging at Crack Depth #1

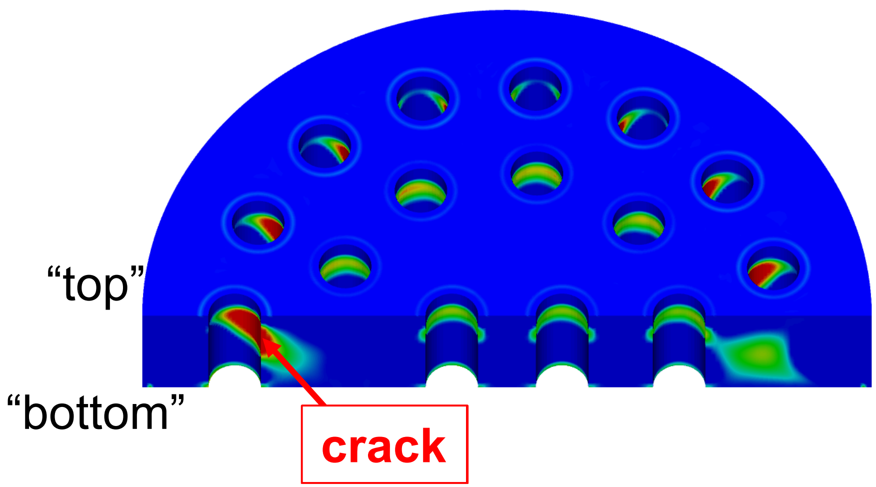


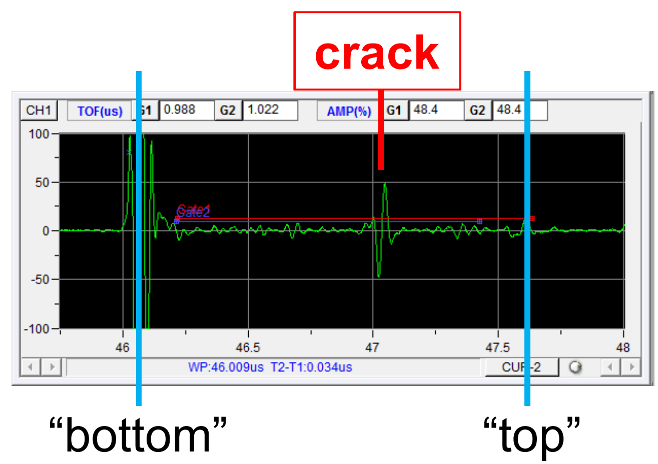
Imaging at Crack Depth #2



Imaging at Crack Depth #3

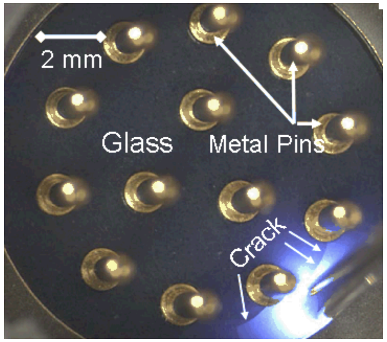


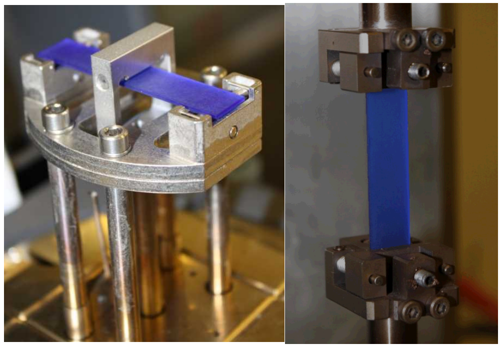


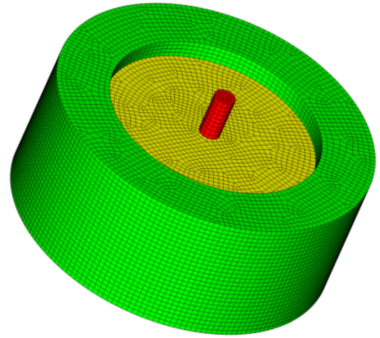


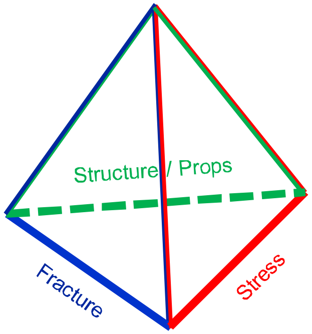
Conclusions

- SPEC model can predict glass response with engineering accuracy
- FEA predictions and data suggest that even short amounts of time may be changing the stress state of the hermetic seal.
- More experiments are necessary to validate long term aging predictions of the SPEC model.
  - FEA can help determine what experiments will be most useful.









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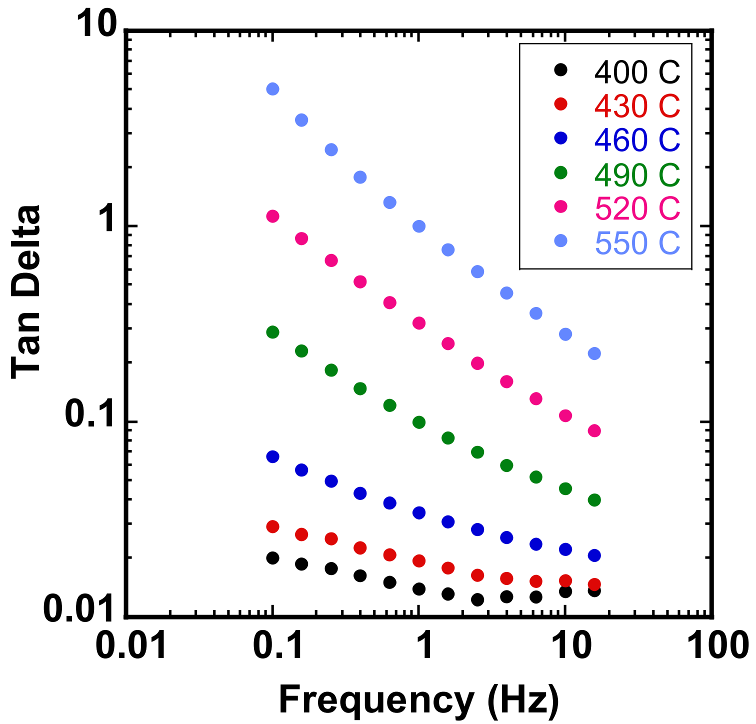
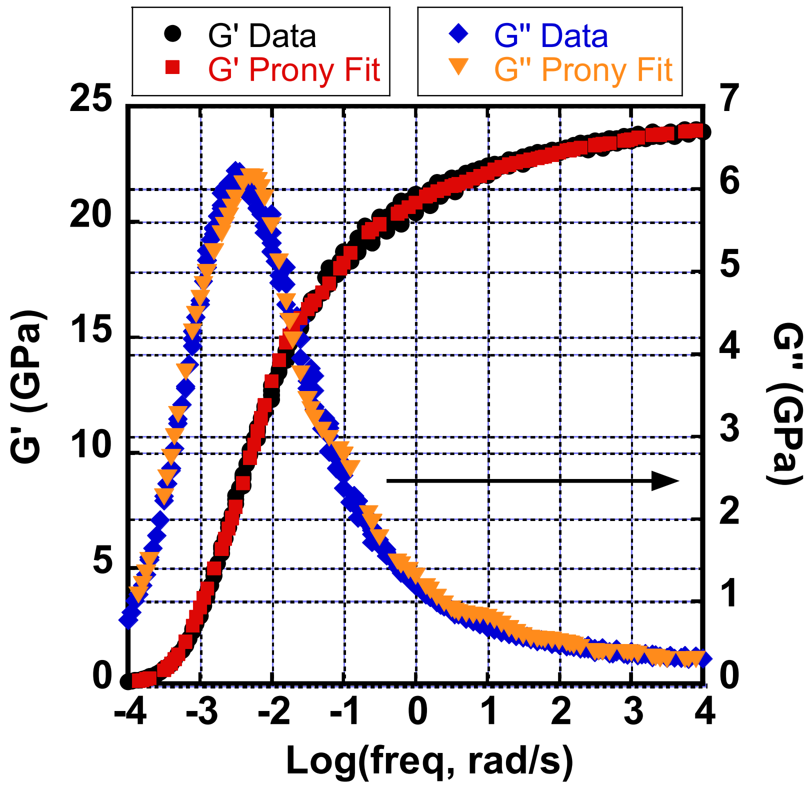

Tan Delta
Frequency (Hz)
400 C
430 C
460 C
490 C
520 C
550 C
10
1
0.1
0.01
0.01
0.1
1
10
100


G' Data
G' Prony Fit
G" Data
G" Prony Fit
G' (GPa)
G" (GPa)
Log(freq, rad/s)
25
20
15
10
5
0
7
6
5
4
3
2
1
0
-4
-3
-2
-1
0
1
2
3
4